

LIST OF U.S. CUSTOMS LABORATORY METHODS

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25-24	ASTM E 815	<u>Test Method for Calcium Fluoride in Fluorspar by Complexometric Titration</u>
25-25	ASTM D 50 <u>NHM - 1994</u>	<u>Test Methods for Chemical Analysis of Yellow, Orange, Red and Brown Pigments Containing Iron and Manganese</u>
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USCL METHOD 25-01

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Mineral and Mineral Products

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

0 INTRODUCTION

The geological identification of building stone is needed for proper tariff classification. U.S. Customs has ruled that the geological definition of marble (HQ 085266 dated 09/20/89) be used by Customs Officers when classifying this merchandise. It is also necessary to determine whether or not an article purported to be a natural stone is in fact synthetically produced and if it contains a resin, thus, making it an artificial stone (HQ 956098 dated 05/16/94). This method is concerned with the analysis of sedimentary, metamorphic, and igneous rocks and minerals. This method discusses observation, microscopic examination, and instrumental techniques used in the analysis of geologic samples for proper identification.

1 SCOPE

The procedures discussed in this method involve instrumental analysis (principally X-ray powder diffraction and fluorescence), thin section analysis using polarizing micro-scopic techniques, observations, and spot tests. The spot test

procedure and observations are normally used in field work and in screening samples in the laboratory. These techniques are valuable in segregating geologic samples into rock types, i.e. sedimentary, metamorphic, or igneous.

Nine procedures are described:

- 1) Sample Screening
- 2) Staining
- 3) Hardness Testing
- 4) Specific Gravity Analysis
- 5) X-ray Powder Diffraction
- 6) X-ray Fluorescence
- 7) Petrographic Analysis
- 8) Refractive Index Determination
- 9) Artificial Stone Analysis

2 GENERAL

The procedures stated here are meant to be used as a guide for analysts in identifying rock and mineral samples. They deal with practices used by scientists when examining rock and mineral samples and it is felt that if the principles listed in this method are followed, a great majority of geologic samples will be correctly identified. It is important for the analyst to carefully examine the sample and make notations before cutting and grinding.

3 REAGENTS AND MATERIALS

- 3.1** Dickson's Stain
Prepare two staining solutions:

- 3.1.1** Solution A: Alizarin Red S concentration of 0.2 g/100 ml of alizarin red in dilute hydrochloric acid (15 ml of concentrated acid made up to 1 liter with water).
- 3.1.2** Solution B: Potassium ferricyanide concentration of 2 g/100 ml of potassium ferricyanide in dilute hydrochloric acid (dilute the acid as in Solution A).
- 3.1.3** Mix solutions A and B in the proportion 3 parts by volume of A to 2 parts of B. Note: The solution of Alizarin Red S in acid may be made up beforehand and will keep, but the potassium ferricyanide must be made fresh each time.
- 3.2** Dilute Hydrochloric Acid (HCl)
Add 20 ml of reagent grade hydrochloric acid to enough water to make a final volume of 1 liter.
- 3.3** Refractive Index Oils
A range of 1.400 - 2.000 is sufficient for geologic materials.

4 APPARATUS

- 4.1** Diamond and agate mortar and pestle. (Mortar)
- 4.2** Hand held lens (usually 10X or 30X). (Lens)
- 4.3** Analytical balance, or a "Jolly Balance."
- 4.4** X-Ray Powder Diffractometer (XRD) with a current set of diffraction data published by the International Centre for Diffraction Data (ICDD).
- 4.5** Infrared Spectrophotometer (IR).
- 4.6** X-Ray Fluorescence (XRF) or equivalent for qualitative elemental analysis.

- 4.7** Diamond cut-off saw with a blade suitable for petrographic sampling.
- 4.8** Grinding and polishing equipment; for rough grinding, a rotating grinder suitable for petrographic work; for fine grinding, a petrographic hand grinder or equivalent.
- 4.9** Abrasive papers: 320, 400, and 600 grit for rough grinding.
- 4.10** Alumina slurry 5-10 microns and polishing cloths.
- 4.11** Polarizing Light Microscope (PLM).

5 SAMPLING AND SAMPLES

- 5.1** Laboratory Sample
The sample received in the Customs laboratory is from a shipment of imported merchandise, therefore, the analyst does not have an opportunity to study the geologic region where the sample originated. Often it is extremely difficult to make determinations such as whether a specimen is metamorphic or sedimentary based on one small sample.
- 5.2** Test Sample
The sample taken for individual testing in the laboratory should be representative of the sample received. Screening and observations should be made prior to any cutting or grinding operations.

6 PROCEDURE

- 6.1** Sample Screening
- 6.1.1** Visually examine the sample for evidence of foliation, fossils, and other features which may aid in classifying as sedimentary, metamorphic, or igneous.
- 6.1.2** Also note whether or not the specimen is polished and if bevels are present.

- 6.2 Staining**
The sample should not be ground so that the analyst can study the reaction of the stain (or acid) on the surface of the sample.
- 6.2.1** Place a few drops of Dickson's stain (or dilute hydrochloric acid) on the surface of the sample.
- 6.2.2** A carbonate mineral such as limestone or marble will effervesce rapidly when treated with the hydrochloric acid solution. Dolomite will also react, but at a much slower rate than limestone or marble. Silicate minerals will not react with hydrochloric acid.
- 6.2.3** The following is a guide to be used for making a preliminary determination using Dickson's stain:

Sample	Stain/HCl Reaction
Carbonates (marble, limestone, travertine)	Red/bubbles (rapid)
Dolomite	Red/bubbles (slow)
Basalt, and other non-carbonaceous minerals	No reaction

- 6.3 Hardness Testing**
- Hardness testing is based on the Moh's hardness system which rates the relative hardness of individual minerals from the softest to the hardest. The softest (talc) is given the number 1 and the hardest (diamond) is given the number 10. The following substances are most commonly tested for hardness: mica (2 - 2.5), calcite (3), dolomite (4), serpentine (4), potassium feldspar (5.5 - 6), and quartz (7). There are two methods used for determining hardness:

- 6.3.1.1** A penny, a glass slide, and a good quality triangular file can be used to evaluate

hardness. They have hardness values of 3, 5 - 5.5, and 6 - 6.5, respectively. The file should be broken so that a triangular corner of the ridged portion can be used for scratching the sample.

- 6.3.1.2** Scratch the surface of the penny with the test sample then wipe away the powdered residue.
- 6.3.1.3** Observe the scratch (if one is present). If a deep scratch is easily made, the hardness is well in excess of 3. Likewise if a mark is barely (or not at all) visible, the hardness is close to 3. If the test sample can be scratched using a fingernail the hardness is 2 or less. If the test sample scratches the penny easily, then use the glass slide. If the sample scratches the slide easily, then it is one of the relatively few minerals having a hardness value of 7 or more. Next scratch the test sample using the file. If only faint scratches appear the hardness is 5.5 - 6. If the penny scratches, but the glass slide does not, the hardness is approximately 4.

- 6.3.2** Hardness pencils are commercially available through earth science supply houses. The pencils are used to scratch the test sample to determine the appropriate hardness value. Hardness values for all minerals are given in mineralogy books.

- 6.4 Specific Gravity**
Specific gravity is necessary for the provisions specified in Chapter 25 of the Harmonized Tariff System (HTS) covering marble, travertine, or other calcareous building stone. (It is also useful in separating and identifying minerals; for example, the specific gravity of calcite is 2.72 and that of dolomite is 2.85.) There is a listing of specific gravity values for various minerals in the *Manual of Mineralogy*, page 564. Travertine is formed by the

precipitation of calcium carbonate from solutions of hot mineral water, therefore, it can be very porous and not yield a value that is consistent. Other analytical techniques such as microscopical analysis will be more definitive.

6.4.1 The sample taken should be from an area that is homogeneous. The sample should not contain any cracks or cavities where air bubbles could form causing erroneous determinations.

6.4.2 Weigh approximately one cubic centimeter of the sample in air (Wa).

6.4.2 Weigh the sample in water (Ww).

6.4.3 The calculation is as follows:

$$\frac{W_a}{W_a - W_w} = \text{Specific Gravity}$$

6.5 X-Ray Diffraction

6.5.1 Grind a representative portion of the sample in a mortar (or a grinding apparatus such as a shatter box).

6.5.2 After grinding to a fine powder transfer the sample to a sample holder.

6.5.3 Care must be taken in tapping the sample into the holder using a razor blade. Preferred orientation may occur. There are other acceptable techniques such as mounting the sample on tape or using silica gel. Each analyst should use the technique which gives them the best result.

6.5.4 Testing parameters such as voltage and milliamperage settings vary with the make of the instrument used. The analyst should consult the manual of the instrument being used for the recommended settings.

6.5.5 Samples composed of essentially one

compound can be identified by using the ICDD file.

6.6 X-Ray Fluorescence

In instances where there are several minerals present or if the analyst has difficulty in identifying the sample, it may be beneficial to perform a qualitative analysis in order to determine the elements present.

6.7 Polarizing Light Microscopy

When analyzing complex systems such as igneous rocks it becomes necessary to separate the various minerals in order to identify the type of rock. It is then that microscopic techniques involving thin section examination and refractive index determinations are used.

6.7.1 Prepare a thin section by cutting a one inch square approximately 1/8 inch thick using the diamond saw.

6.7.2 Mount the specimen to a labeled glass slide using epoxy. Press and level the sample on the slide and allow to cure. Care must be taken when preparing the epoxy and mounting the sample to ensure that air bubbles are not present.

6.7.3 Place the slide in a slide holder and grind the sample using the grinding papers until a thickness of approximately 50 microns is reached.

6.7.4 After rough grinding, the sample is then cleaned in an ultrasonic cleaner.

6.7.5 Fine grind the sample to 30 microns using the alumina slurry in distilled water. Instructions for diluting are located on the bottle containing the alumina. Use a hand polisher with a nylon or silk polishing cloth (3). The slide is then ready for PLM analysis.

6.7.6 The analyst should take careful notes of observations made and where possible take photomicrographs and carefully

document the photographs (magnification, whether crossed polarizers were used, etc.).

- 6.8** **Refractive Index Determination**
This method is not applicable to carbonate rocks. Mineralogy books list refractive indices of minerals along with other properties, such as chemical composition, etc. The method calls for using a refractive index oil with a low value and one with a high value, then systematically narrowing the gap until the correct value of the unknown is reached. Care must be taken that a single compound of homogeneous crystallinity is used.
- 6.8.1** Grind a portion of the sample using a mortar and pestle. (If the sample is hard and relatively large, it may be necessary to break it into small pieces using a diamond mortar.)
- 6.8.2** If the mineral is a complete unknown, select an oil in the middle of the range. Place a few crystals on a glass slide and allow a drop of the oil to fall on the crystals, being careful not to touch the crystals to the dropper as this will contaminate the dropper and thus contaminate the oil. When using this method one must be sure to mark on the slide the value of the refractive index oil which is being used.
- 6.8.3** Place the slide under a microscope and bring the specimen into focus.
- 6.8.4** As the stage is lowered, a bright line will appear to surround the crystal. This line will move either into the sample or the surrounding medium. (This line is referred to as the "Becke line.") When the stage is lowered and the line moves into the crystal, then the crystal has a higher refractive index than the oil. If the line moves into the oil, the oil has a higher refractive index than that of the

crystal. Observe the crystal, if it appears to have a clearly defined sharp outline and a great deal of relief, then the index of refraction of the oil is much different than the crystal.

- 6.8.5** After a starting point has been established, place crystals on several glass slides and repeat the procedure. When the indices of refraction of the oil and the crystal are equal the crystal will appear to disappear under magnification.
- 6.9** **Artificial Stone Analysis**
If a sample is suspected of containing an organic binder separating the organic compound from the inorganic can be accomplished by using pyrolysis.
- 6.9.1.1.** Grind a portion of the sample in a mortar and place it in a small test tube.
- 6.9.1.2** Heat the test tube over a flame and allow the fumes from the binder to condense on a KBr (potassium bromide) or suitable disk for IR analysis.
- 6.9.2** The sample may also contain an inorganic binder. Identification may be difficult as the material may closely resemble natural breccias or conglomerates. The first step is to identify the aggregate particles as their compositions and weights must be subtracted when quantitative determinations of an inorganic binder are called for. Aggregate compositions may be determined by the means already discussed such as thin section analysis. Thin sections are also useful in determining the composition of the fines intimately mixed with the binder. Binders are usually cementitious and give off large quantities of water when heated in a test tube. While carbonate rock natural breccias contain abundant water, their cementing material is ordinarily banded. In addition the natural breccias cementing material is composed of calcite which is

identifiable by thin section. Natural volcanic breccias are anhydrous. Natural conglomerates contain rounded aggregate particles, but may contain various binder materials which may be determined by XRD or thin section analysis. Natural brecciated serpentines are cemented with crystalline calcite. Cured portland cement contains portlandite (calcium hydroxide) which can be identified by XRD. It is also characterized by high calcium content as determined by XRF. Pozzolan and slag cements may or may not contain portlandite, but they contain alumina and higher silica percentages than portland cements. Aluminous cements contain very little silica. Sorel cements are characterized by high magnesium and low calcium contents.

A System of Mineralogy, Vol. 1,
7th Edition,
J. D. Dana
John Wiley & Sons, New York, 1944

*Atlas of Sedimentary Rocks Under The
Microscope*,
A. E. Adams, W. S. Mackenzie, and C. Guilford
John Wiley & Sons, New York, 1984

Petrography,
Williams, Turner, and Gilbert,
W. H. Freeman, San Francisco, 1954

Polarized Light Microscopy,
McCrone and Delly
McCrone Research Institute, Chicago, 1987

7 BIBLIOGRAPHY

Manual of Mineralogy, 20th Edition
Cornelius Klein and Cornelius S. Hurlbut, Jr. John
Wiley & Sons, New York, 1985

Note: There are several references cited
at the end of each chapter.

Optical Mineralogy, 3rd Edition
Paul Kerr, McGraw Hill, 1959

Buehler Digest, Vol. 24 No. 1

Crystal Structures of Minerals,
L. Bragg and G. F. Claringbull
G. Bell & Sons, Ltd., London, 1965

*An Introduction to The Rock Forming
Minerals*,
Deer, Howie, and Zussman
John Wiley & Sons, New York, 1966

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USCL METHOD 25-02

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Fluorspar Analysis

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

Fluorspar is provided for in Chapter 25 of the Harmonized Tariff Schedule of the United States (HTSUS). Classification is dependent upon the weight percent of calcium fluoride present. The recommended U.S. Customs Method for the determination of the weight percent of calcium fluoride present in fluorspar is the method given in the Certificate of Analysis issued for the National Institute of Standards and Technology (NIST) Standard Reference Material 79A - Fluorspar.

2 REFERENCES

*Certificate of Analysis
National Institute of Science and
Technology Standard Reference
Material 79A - Fluorspar
National Institute of Science and
Technology*

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USCL METHOD 25-03

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Hydraulic Cements

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

Portland cement, aluminous cement, slag cement, supersulfate cement and similar hydraulic cements, whether or not colored or in the form of clinkers, are classified in Chapter 25 of the Harmonized Tariff Schedule of the United States. Admixtures of any aggregate material does not fall under this heading and HTSUS 3824 should be considered.

2 REFERENCES

ASTM C 114

Standard Test Methods for Chemical Analysis of Hydraulic Cement

ASTM C 150

Standard Specification for Portland Cement

3. PRINCIPLE

By incorporating the referenced method and specifications, different types of hydraulic cement can be distinguished.

4 APPARATUS, REGENTS AND MATERIALS

4.1 Energy dispersive X-ray spectrometer system with a fundamental parameters program for XRF analysis

4.2 X-ray diffraction system capable of scanning between ten (10) and eighty-five (85) degrees 2-Theta

4.3 Regents, materials and apparatus as mentioned in ASTM C114 if chemical analysis is necessary.

5. PROCEDURE

5.1 Sample Description

The sample should be uniform in both color and particle size. Multiple colors and/or particle sizes indicate the sample was blended after grinding and probably should not be classified in Chapter 25. Clinkers are an exception to this as they will come in numerous sizes and shapes.

5.2 Test for Hydraulic Cement

5.2.1 Mix a portion of the sample with water to form a thick paste. Divide into two sections.

5.2.2 Let one portion sit exposed to air for forty-eight (48) hours. If it sets up, the sample is cement.

5.2.3 Cover one portion with room temperature water. Let it sit for forty-eight (48) hours. If it sets up, the sample is hydraulic cement.

5.3 Determination of Composition

Composition determines the type of hydraulic cement. See references for composition information.

5.3.1 X-ray Diffraction

Prepare sample as required by the specific instrumentation. Run according to standard conditions as specified by the manufacturer and required by the instant sample.

5.3.2 X-ray Fluorescence

Prepare sample as required by the specific instrumentation. Run according to standard conditions as specified by the manufacturer and required by the instant sample.

5.3.3 Refer to ASTM C114 if chemical analysis is necessary.

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Recommended Guidelines for Inorganic Qualitative and Quantitative Analysis

SAFETY PRECAUTION

This method does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 SCOPE AND FIELD OF APPLICATION

The following list of references contains procedures which should prove useful in the qualitative and quantitative analysis of inorganic materials of Chapter 25, 26, 28, 31,32 and 38 HTSUS. This list is being provided for general guidance and should not be considered exhaustive.

2 REFERENCES

Determination of Manganese in Manganese Ore, in **Scott's Standard Methods of Chemical Analysis**, 5th ed.
N.H. Furman
Van Nostrand, New York 1939

Procedure for Copper in Ores, in **Scott's Standard Methods of Chemical Analysis**, 5th ed.
N.H. Furman
Van Nostrand, New York 1939

Determination of Lead in Ore as Lead Chromate, in **Scott's Standard Methods of Chemical Analysis**, 5th ed.
N.H. Furman
Van Nostrand, New York 1939

Zinc Titration in Alkaline Solution, in **Scott's Standard Methods of Chemical Analysis**, 5th ed.
N.H. Furman
Van Nostrand, New York 1939

Determination of Tungsten in Ore and Concentrates, in **Scott's Standard Methods of Chemical Analysis**, 5th ed.
N.H. Furman
Van Nostrand, New York 1939

Nickel Determination by the Dimethylglyoxime Method, in **Standard Methods of Chemical Analysis**, , 6th ed.
N.H. Furman and F.J. Welcher
Van Nostrand, New York 1963

Vanadium by Precipitation with Lead Acetate, in **Standard Methods of Chemical Analysis**, 6th ed.

N.H. Furman and F.J. Welcher
Van Nostrand, New York 1963

Molybdenum Determination by
Precipitation as Lead Molybdate, in
***Standard Methods of Chemical
Analysis***, 6th ed.

N.H. Furman and F.J. Welcher
Van Nostrand, New York 1963

Sulfur, Sublimed or Precipitated, in
USP

United States Pharmacopeial
Convention Inc.
Rockville, MD, 1994

Iodine, in ***USP***

United States Pharmacopeial
Convention Inc.
Rockville, MD, 1994

Silica Gel, in ***USP***

United States Pharmacopeial
Convention Inc.
Rockville, MD, 1994

Precipitated Calcium Carbonate, in
USP

United States Pharmacopeial
Convention Inc.
Rockville, MD, 1994

Determination of Copper, in ***Rapid
Quantitative Electrolytic Methods
of Analysis***, 7th ed.

George W. Slomin

E.H. Sargent & Co., Chicago
1947.

McGraw-Hill, New York, 1990

Spot Tests in Inorganic Analysis

F. Feigl and V. Anger
Elsevier Publishing Co.,
Amsterdam,
1972

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ASTM C 561 NHM - 1991 Test Method for Ash in Graphite

2 REFERENCES

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

ASTM C561

Test Method for Ash in a Graphite Sample

0 INTRODUCTION

This method provides a quick and practical means of determining the ash content of graphite. It is usually run consecutively with **ASTM C562** for moisture in graphite. The method is widely used to determine the ash content of most carbon products.

1 SCOPE AND FIELD OF APPLICATION

The ash content of graphite is the primary determinant of the purity of the sample. High purity is normally associated with artificially produced graphite of HTSUS 3801.

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ASTM E 360 Test Methods for Chemical Analysis of Silicon and Ferrosilicon - Modified

ASTM E 360

Test Methods for Chemical Analysis
of Silicon and Ferrosilicon - Modified

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This method is designed for the analysis of ferrosilicon or silicon metal containing a restricted range of alloyed metals or impurities. It does not sufficiently address the determination of iron oxide, and the silicon analysis involves the use of perchloric acid for dehydration. It may be useful in the analysis of commodities covered by Subheadings 2505.10.10 and 2506.10.00.10 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

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ASTM C 146 Test Methods for Chemical Analysis of Glass Sand

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

0 INTRODUCTION

ASTM C146 provides several methods of determining silica and iron oxide as required by Headings 2505.10.10 and 2506.10.00.10 of the Harmonized Tariff Schedule of the United States (HTSUS). The method involving the determination of silica by treating with H_2SO_4 - HF is probably the most suitable. Iron is determined by a photometric method but this could be revised to employ current instrumental methods.

1 SCOPE AND FIELD OF APPLICATION

This method is applicable not only to glass sands but to any sand composed predominantly of silica. It is adaptable to varying levels of Si_2O_3 .

2 REFERENCES

ASTM C 146

Test Methods for Chemical Analysis of Glass Sand

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ASTM C 837

Test Method for Methylene Blue Index of Clay

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This test method covers the measurement of the adsorption of methylene blue dye by a clay material which is then calculated as the "methylene blue index" of the clay. The method is to be used as appropriate in Chapter 25 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

ASTM C 837

Test Method for Methylene Blue Index of Clay

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ASTM C 199

Test Method for Pier Test for Refractory Mortars - Modified

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This method is applicable to refractory mortars of Heading 3816 in the Harmonized Tariff Schedule of the United States (HTSUS). Some of the ingredients of refractory mortars are included within HTSUS Chapter 25 but they would not be tested mechanically as a finished mortar would. This method is to be used when appropriate in the analysis of commodities covered by the HTSUS.

2 REFERENCES

ASTM C 199

Test Method for Pier Test for Refractory Mortars - Modified

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Recommended Guidelines for Analysis of Clay

SAFETY PRECAUTION

This method does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 SCOPE AND FIELD OF APPLICATION

The following list of references contains procedures which should prove useful in the analysis of clay provided for in Chapter 25 HTSUS. This list is being provided for general guidance and should not be considered exhaustive.

2 REFERENCES

Clay and Calcined Clay DTA Methodology, in
Laboratory Manual of Petrographic Techniques
C.S. Hutchinson
John Wiley & Sons, New York ,
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Firing Properties of Clay in
Applied Clay Mineralogy
R.E. Grim
McGraw Hill, New York, 1962

Clay Minerals: A Guide to Their X-

Ray Identification,
Dorothy Carroll in
Geology Society of America, Special Paper 126, 1970

Identification of Clay Mineral by Staining Tests,
R.C. Mielenz and M.E. King in
ASTM Proceedings, Vol. 51, 1951

Analysis of Clay in
Scott's Standard Methods of Chemical Analysis, Vol. II
N.H. Furman, ed. Van Nostrand, New York, 1939.

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-11

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ASTM C 373 **Test Method for Water Absorption, Bulk Density,** **Apparent Porosity and Apparent Specific Gravity** **of Fired Whiteware Products - Modified**

This is a duplicate method. Please see
USCL 69-03.

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

2 REFERENCES

ASTM C 373

Test Method for Water Absorption,
Bulk Density, Apparent Porosity and
Apparent Specific Gravity of Fired
Whiteware Products - Modified

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-12

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ASTM C 119 Scientific Definition of Granite

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

0 INTRODUCTION

The text provides general information regarding building stones. Most particularly it provides a “scientific definition of granite” which is based on the principles of the IUGS (International Union of Geological Sciences) classification system, an internationally recognized system based on relative percentages of major mineral components. Correct granite classification is required for Headings 2516 and 6802 of the Harmonized Tariff Schedule of the United States (HTSUS).

1 SCOPE AND FIELD OF APPLICATION

The parameters for specifying a granite classification are somewhat more liberal than those of the IUGS system. This is desirable as it will tend to cover unavoidable statistical inaccuracies encountered in granite sampling.

2 REFERENCES

ASTM C 119

Scientific Definition of Granite

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-13

Index

ASTM D 8 **Terminology Relating to Materials for Roads** **and Pavements**

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

The text consists of a glossary defining various materials used in road building and related construction projects. It may be of assistance in understanding the makeup and classification of the products of Heading 2517 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

ASTM D 8

Terminology Relating to Materials for Roads and Pavements

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-14

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ASTM C 11 Definition of Terms Relating to Gypsum

2 REFERENCES

ASTM C 11

Terminology Relating to Gypsum and
Related Building Materials and
Systems

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

0 INTRODUCTION

The text consists of a vocabulary listing of commonly used gypsum-based building material terms. Included is a list of ASTM referenced documents in which specifications for many types of gypsum products are given.

1 SCOPE AND FIELD OF APPLICATION

ASTM C11 is useful primarily in providing background in gypsum industry terminology and is helpful in understanding and interpreting the texts of other ASTM documents relating to gypsum. Some terminology concerns compositions of gypsum plasters of Subheading 2520.20 of the Harmonized Tariff Schedule of the United States (HTSUS).

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USCL METHOD 25-15

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ASTM C 22 Specifications for Gypsum

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This reference contains information regarding the specification of gypsum. This information may be useful in Chapter 25 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

ASTM C 22
Specifications for Gypsum

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-16

Index

ASTM C 26 Methods of Testing Gypsum and Gypsum Products

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This method is used in the analysis of gypsum and gypsum products. It is to be used as applicable in the analysis of commodities of Chapter 25 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

ASTM C 26

Methods of Testing Gypsum and Gypsum Products

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-17

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ASTM C 28 Specifications for Gypsum Plasters

2

REFERENCES

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

ASTM C 28

Specifications for Gypsum Plasters

1 SCOPE AND FIELD OF APPLICATION

This specification includes:

- a) a terminology of gypsum plaster types
- b) a minimum requirement of 66 percent $\text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O}$ which is to be determined by **ASTM C471**.
- c) a table of permissible aggregate types and concentrations for the different plaster grades.
- d) a method of determining weight and volume ratios of aggregate to plaster.

From the information so obtained, one can determine if a plaster meets the qualifications of one of the plaster grades given. Qualification as a plaster is required by Subheading 2520.20 of the Harmonized Tariff Schedule of the United States (HTSUS).

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USCL METHOD 25-18

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ASTM C 59 Specifications for Gypsum Molding Plaster

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

ASTM C 59

Specifications for Gypsum Molding Plaster

1 SCOPE AND FIELD OF APPLICATION

This specification refers to both gypsum casting plasters and gypsum molding plasters. Permissible properties such as chemical composition fineness, setting time, and compressive strength are given and references of analytical methods to obtain these data are proposed. Meeting of these specifications is required to qualify as a plaster of Subheading 2520.20 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-19

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ASTM C 61 Specifications for Keene's Cement

ASTM C 61

Specifications for Keene's Cement

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This specification is useful in defining Keene's cement by physical properties. Acceptable setting times, compressive strength, and fineness for Keene's cement are given and references to corresponding test methods are given. Samples qualifying as Keene's cement are classified under Subheading 2520.20 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-20

Index

ASTM E 20

Practice for Particle Size Analysis of Particulate Substances in the Range of 0.2 to 75 Micrometers by Optical Microscopy

Microscopy

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This is a highly detailed and corrected method for determining particle sizes. As stated in the text, the procedures are too long and too involved to be used on a routine basis but they are quite suitable for calibration and are particularly useful for measuring irregular particles. A number of tips on obtaining better particle dispersion are given. It may be useful in the analysis of commodities covered in Chapter 25 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

ASTM E 20

Practice for Particle Size Analysis of Particulate Substances in the Range of 0.2 to 75 Micrometers by Optical

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-21

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ASTM D 2131 Specification for Natural Muscovite Mica Splittings

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This specification provides a classification system of thirteen grades based upon size, form, and splittings thickness. The grades are then qualified by meeting minimum defects requirements as determined by a counting method. This method appears to be an attempt to classify mica splittings under Subheading 2525.10 of the Harmonized Tariff Schedule of the United States (HTSUS) on an organized, less subjective system than the Bengal Indian System.

2 REFERENCES

ASTM D 2131

Specification for Natural Muscovite
Mica Splittings

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-22

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ASTM D 351

Classification for Natural Muscovite, Block Mica and Thins Based on Visual Quality

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

2

REFERENCES

ASTM D 351

Classification for Natural Muscovite, Block Mica and Thins Based on Visual Quality

1 SCOPE AND FIELD OF APPLICATION

This method is based on the Bengal India Grading System. It is an arbitrary system but, at this time it is the one that prevails. Included are a number of mica grade definitions and corresponding thicknesses which are required for classifications in Subheading 2525.10.00 of the Harmonized Tariff Schedule of the United States (HTSUS).

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USCL METHOD 25-23

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ASTM D 374

Test Methods for Thickness of Solid Electrical Insulation

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This method discusses the operation and calibration of instruments for measuring the thickness of solid electrical insulation. For the calibrated instruments could be used to measure mica thicknesses so as to distinguish between split block mica, mica splittings, and other grades of Subheading 2525.10.00 of the Harmonized Tariff Schedule of the United States (HTSUS).

2 REFERENCES

ASTM D 374

Test Methods for Thickness of Solid Electrical Insulation

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-24

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ASTM E 815 Test Method for Calcium Fluoride in Fluorspar by Complexometric Titration

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

2 REFERENCES

ASTM E 815

Test Method for Calcium Fluoride in Fluorspar by Complexometric Titration

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-25

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ASTM D 50

NHM - 1994

Test Methods for Chemical Analysis of Yellow, Orange, Red and Brown Pigments Containing Iron and Manganese

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

The text describes wet chemical test methods used to determine percentages of iron oxides and other components of Heading 2530 of the Harmonized Tariff Schedule of the United States (HTSUS). The method can be used to distinguish between iron oxide materials of HTSUS Heading 2530 and HTSUS Heading 2821.

2 REFERENCES

ASTM D 50

Test Methods for Chemical Analysis of Yellow, Orange, Red and Brown Pigments Containing Iron and Manganese

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-26

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Recommended Guidelines for Analysis of Rock and Mineral Samples

Blackwell Scientific Publications,

SAFETY PRECAUTION

This method does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Oxford, 1989

Modal Analysis Estimation Methods and Staining Tests for Igneous and Metamorphic Rock, in
Laboratory Manual of Petrographic Techniques

C.S. Hutchison
John Wiley and Sons, New York,
1974

1 SCOPE AND FIELD OF APPLICATION

The following list of references contains procedures and references which should prove useful in the analysis of rock and minerals provided for in Chapter 25 HTSUS. This list is being provided for general guidance and should not be considered exhaustive.

The Sampling and Analysis of Crude, Caustic, and Dead-Burned Magnesite, in
Scott's Standard Methods of Chemical Analysis, Vol. I

N.H. Furman, editor
Van Nostrand, New York, 1939

2 REFERENCES

Glossary of Terms and Explanation of the IUGS Classification System for Igneous Rocks, in
A Classification of Igneous Rocks and Glossary of Terms, International Union of Geological Sciences (IUGS)
R.W. LeMaitre, editor

U.S. CUSTOMS LABORATORY METHODS

USCL METHOD 25-27

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ASTM D 2488

NHM - 1993

Practice for Description and Identification of Soils (Visual-Manual Procedure)

SAFETY PRECAUTIONS

This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 SCOPE AND FIELD OF APPLICATION

This is a multifaceted system based on the physical characteristics of soil. The tests require sieves but little else in the way of equipment. However, expertise is only gained by experience.

2 REFERENCES

ASTM D 2488

Practice for Description and Identification of Soils (Visual-Manual Procedure)